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X-RAY STUDY OF AGAVE VERA CRUZ FIBRE

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Plate VI A & B

ABSTRACT. The structure and properties of Agave vera cruz have been studied with the help of X-ray diffraction analysis. This particular type of leaf fibre, which is used by local weavers in Burma for making longyi, has not so far received due attention as far as its structural study by X-ray method is concerned. The length of the (002) arcs has been found to be greater than that in any bast fibre. It has also been found that, unlike cotton, the strength of the fibre decreases with the decrease in spiral angle and also that the strength of the fibre decreases with the increasing concentration of mercerising solution. An explanation of this phenomenon has been incorporated in the paper

1. INTRODUCTION

It is well known that cotton occupies a unique position in the family of natural textile fibres. Next comes jute which belongs to the 'bast fibre' group. Another group classed as 'leaf fibres' is a comparatively newcomer in the domain of textile industry. Till now, leaf fibres are mainly used in cor industry. The bare fact they have not made their mark as quality textile fibres in the past does not necessarily rule out the possibility of their economic utilisation in the future. In fact, a particular type of leaf fibre—Agave vera cruz, which finds its use in longyi industry in Burma, forms the subject matter of our present investigation

An exhaustive study of natural fibres has already been made by many investigators (Meyer and Mark 1928; Sirkar and Saha 1944; Sisson, 1943). The results of the investigations are so well known that a resume here may appear redundant Nevertheless, some salient features deserve mention. The chains of which a fibre is built may lie either closely parallel to or a long a spiral path around the fibre axis. It was later suggested that all natural fibres have their crystallites arranged in spiral manner. It should be pointed out that a very steep spiral amounts almost to parallel configuration. In ramie, crystallites are arranged in a very steep spiral form, *i e.* in parallel configuration. This so called parallel orientation, howsoever perfect from crystallographic point of view, is not an ideal configuration so far as textile properties of a fibre are concerned It is true that this perfect alignment of the crystallites in ramie provides to the fibre very good strength and rigidity, but there is an associated brittleness in the transverse direction. In cotton, the crystallites are arranged in spirals making an angle of about 30° with

the fibre axis. Though cotton has less intrinsic strength than ramie, it is more resilient and weaves into a softer fabric.

The degree of steepness of a spiral finds its manifestation in the length of the (002) arcs. A cellulose fibre having crystallites arranged in steep spirals will give an X-ray diagram having well defined spots. But as the spiral arrangement is more and more flat, *i e.* spiral makes larger angles to the fibre axis, the reflections are drawn out into larger and larger arcs.

The width of the (002) reflection, however, depends on the size of crystallites, the fluctuation in arrangement of crystallites along the spiral and on the presence of constituents other than cellulose in a marked degree. A correct idea of the orientation and the size of the crystallites can, therefore, be obtained from the length and width of the (002) reflections It may be mentioned that the measurement of intensity along different directions of the (002) reflection provides a good method of classification of fibres (Sirkar and Saha, 1946).

It needs hardly any mention that a precise knowledge of the molecular structure of a fibre is essential to have a deeper understanding and clear insight of the properties of a fibre; and the X-ray diffraction analysis is the best available method for the purpose. In view of the fact that the leaf fibres have not yet received any systematic and extensive study by X-ray method, the present investigation on the X-ray study of the structure and properties of Agave vera cruz has been undertaken by us.

2. EXPERIMENTAL

Raw leaves of Agave vera cruz were retted as usual in our laboratory and the fibres thus obtained from the retted leaves were thoroughly washed, dried and combed before taking X-ray photographs The specimens were prepared in the form of a bundle containing about twenty strands, each cut into 2.5 cm in length. The individual fibres in the specimen were made parallel to each other by applying a little tension and the bundle thus made was fixed on a specially made specimen holder with the fibre axis vertical. The specimen was then placed against the X-ray beam collimated through a slit of 0.05 cm in diamter and 6 cm in length. Filtered Cu Kα radiation from a demountable 'Raymax 60' X-ray tube was used all throughout the investigation.

Samples mercerised with 15%, 20%, 25% and 30% NaOH solution at room temperature were also photographed. Two samples with different conditions of pre-treatment were bleached by passing chlorine gas in the fibres kept in water. The physical properties such as fineness and intrinsic strength of the fibres, both raw and treated with mercerising solutions of different strength, were determined.

All X-ray photographs reproduced in this paper were taken with Unicam single crystal goniometer; the film to specimen distance in each photograph being 3 cm.

Calibration was made by taking X-ray pattern on the same film of a copper wire whose spacings are known accurately.

3. RESULTS AND DISCUSSIONS

X-ray patterns of raw and treated Agave vera cruz are reproduced in Plate VI A & B. The pattern due to raw fibre (Fig. 1) corresponds to that of native cellulose, though some differences in finer details are observed. The equatorial reflections $A_1(101)$ and $A_2(10\bar{1})$ are nearly fused together. The $A_1(002)$ arcs, which are most intense, end rather abruptly and continue with weaker intensity along the diffraction ring. The spacings calculated from figure 1 (Plate VI A) are given in Table below.

TABLE I

Spacings in Å	tensity	Planes (*hkl*)
14 45	w.	?
9 57	m.w.	?
5 92	s	101
5 42	s	$10\bar{1}$
3.93	v s.	002
6.35	m.w.	110
5 15	w.	020
4.3	m.s.	120
4.2	m.	?
2 56	m.s.	040

The unit cell dimension calculated from these spacings was found to be quite in agreement with that of native cellulose. It can be seen from the table above that three spacings whose planes are not identified do not belong to cellulose spacings. Figure 1 shows the presence of a few irregular spots scattered all over the diagram. These are due to diffraction by calcium oxalate crystals present in the fibre. Our preliminary chemical analysis indicated the presence of these crystals. Our findings do not support the suggestion made by Kubo (1940) that the cellulose present in Agave fibres is in the modified form, known as Cellulose T

Our chemical analysis shows that the cellulose content of this fibre is about 75% and the next major constituent lignin is about 15%. Two reflections near the central spots of spacings 14 35A and 9.57A (Table I) appear to be due to lignin. It is well known that stronger the caustic soda solution used for mercerisation, the larger is the quantity of lignin removed from the fibres It can be seen from X-ray patterns (figures 5, 6, 7, 8 of Plate VI B) of mercerised fibre that these two reflections get gradually weaker as the strength of mercerising solution is increased. This suggests that these two spacings are due to lignin present in the fibre. Another reflection of spacing 4.2A(Table I), intense on the meridian, seems to be due to

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PLATE VI A

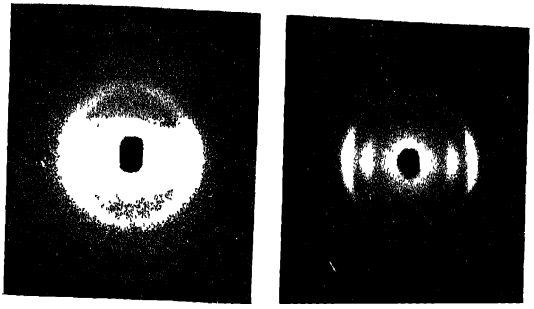


Fig. 2

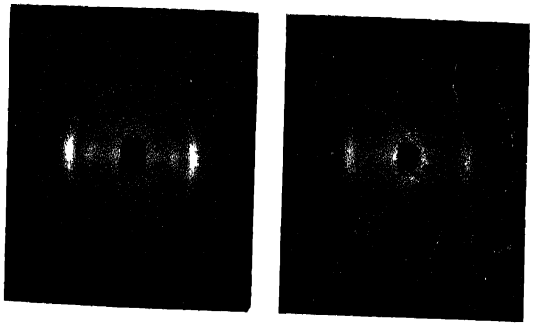


Fig. 4

X-ray diffraction patterns

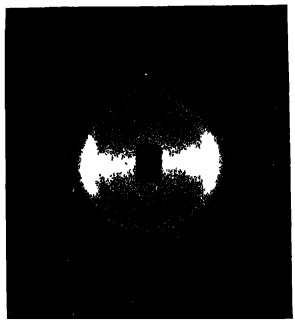
Fig. 1. Raw Agave vera cruz

Fig. 2. Bleached fibre (lignin partially removed)

Fig. 3. Bleached fibre (lignin removed in higher degree)

Fig. 4 Fibre kept in boiling water for one hour

Fig 5



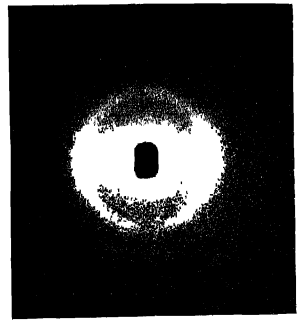
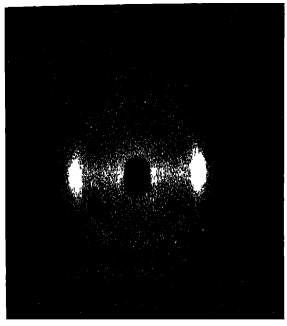


Fig 6

Fig 7



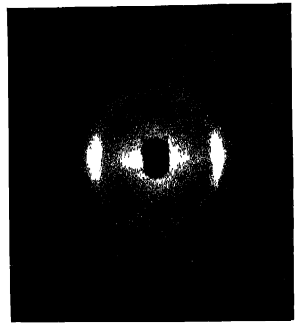


Fig 8

X-ray diffraction patterns

Fig 5 Mercerised with 15% NaOH solution.
Fig. 6 ,, ,, 20% ,, ,,
Fig 7 ,, ,, 25% ,, ,,
Fig 8. ,, ,, 30% ,, ,,

wax in the fibre. That this spacing is due to wax has been confirmed by taking the X-ray photograph of the dewaxed sample where this particular reflection is absent.

All our X-ray photographs show that the (002) reflections are drawn out into arcs which are longer than those due to bast fibres. The length of the arc in the case of raw Agave fibre has been found to be about 60° (figure 1). The extented arcs indicate that the pattern corresponds to cellulose crystallites oriented in spiral form, the spiral making an angle of 30° with the fibre axis Four fainter arcs distributed diagonally on the meridian can also be seen in figure 1. They correspond to (021) reflections and are not as sharp as they are in ramie or flax. The lack of sharpness as well as the extension of the arcs in the present case may be due to the flat spiral configuration A comparison of the (002) arcs in figure 1 with those in the X-ray pattern of cotton showed that the arcs in the former are much wider. This pronounced width suggests that the size of the crystallites in this case is smaller. It also indicates a lower degree of lateral order and a marked fluctuation of the orientation of crystallites around the spiral.

X-ray photographs of two Agave vera cruz samples from which lignin was removed in different degree and bleached thereafter by passing chlorine gas revealed some interesting informations A careful examination of figure 2, which is due to the sample from which the lignin and other intercrystalline materials were removed only partially, shows that the (002) reflection is sharper and the pattern as a whole is cleaner than those due to raw fibre (figure 1) Figure 3, which is due to the sample from which the lignin and amorphous constituents were removed in a higher degree, shows that the pattern (figure 3) is undoubtedly cleaner than figure 2 and the (002) reflection is almost as sharp as it is in cotton which is regarded as almost pure cellulose. It should be noted that the strength of the latter was markedly less than the former (figure 3). This marked loss of strength in the latter sample, therefore, seems to be due to the higher degree of removal of lignin and other intercrystalline constituents A comparative study of figures 2 and 3 also reveals that the reflections due to lignin of spacings 14.35Å and 9.57Å are very poor or nearly absent in figure 3. This finding suggests that lignin helps in retaining the strength of the fibre

It may be observed from the X-ray photograph (figure 4) of fibres kept in boiling water for about an hour that the reflections are sharper and the pattern as a whole is cleaner than those due to raw fibre. It is also to be pointed out that the (020) reflection on the meridian is more clearly visible and the length of the (002) arcs is less than that in raw fibre, i.e. 50°. This reduced length of the (002) arcs and their sharpness indicate better orientation and crystallinity. No change in spacings was, however, observed.

X-ray photographs of samples mercerised with 15%, 20%, 25% and 30% caustic soda solutions are reproduced respectively in figures 5, 6, 7 and 8

(Plate VI B). A careful examination reveals that in all these photographs, in addition to the usual spacings of mercerised cellulose, two reflections of spacings 5.92Å and 5 4Å due to 101 and $10\bar{1}$ planes of the native cellulose are present. This shows that both native and mercerised cellulose (Saha, 1948; Sisson and Sanor, 1941) are present in the samples. The mercerisation is not complete even in the sample treated with 30%NaOH solution. The size of the unit cell calculated from the spacings in the mercerised patterns, given in Table II below, was found to be the same as that in hydrated cellulose (Sirkar and Saha, 1946, Andress, 1929).

TABLE II

Spacings in A	Intensity	Planes (*hkl*)	
7.36		101	
4.4		101	
4.03		002	
5 2		020	
4 35		120, 021	
2 56		040	
5 92		101	Native
5 4		101	Native

In cotton, mercerisation was found to be complete when it was treated with 18% NaOH solution, but in the present case even a 30% solution could not effect complete mercerisation This appears to be due to the predominance of intercrystalline materials present in this fibre which affects mercerisation adversely It may be mentioned that the degree of mercerisation is more pronounced in a sample kept in boiling water before being mercerised The degree of mercerisation in the sample kept in boiling water for about an hour and then mercerised with a 30% NaOH solution has been found to be more complete, as revealed by X-ray examination, than that in the sample which was mercerised as usual with the same concentration of mercerising solution (figure 8). This enhanced degree of mercerisation appears to be due to the swelling—a factor favouring mercerisation—effected by boiling; and also perhaps due to the removal of some intercrystalline constituents which affect mercerisation.

It may be noted that the nature of the diffraction pattern, particularly the (002) arcs, gives an idea of the strength of the fibre. The longer the arcs, the greater is the departure of the orientation of spiral with respect to the fibre axis, which, as is well known, accounts for the loss of strength. Table III below shows the relationship between the intrinsic strength of the fibres, both raw and and

mercerised, and the length of the (002) arcs or the spiral angle. It also shows the relationship between the intrinsic strength and the concentration of the mercerising solutions.

TABLE III

Per cent NaOH	Intrinsic strength $10^6 \frac{\text{gm}}{\text{gm/cm}}$ (strength fineness)	Spiral angle (in degrees)
Raw fibre	2.35	
15 %	1.41	
20 %	1.18	22 5
25 %	×	20
30 %	0.97	

It can be seen that the raw fibre with spiral angle 30° has intrinsic strength 2 35, which means that 23 5 kilometres length of fibre will break by its own weight. It is evident from Table III that as the concentration of the mercerising solutions increases, the intrinsic strength decreases. Table III also shows that the intrinsic strength decreases with the decrease in spiral angle. These observations are in contrary to that observed in cotton, where the intrinsic strength increases with the decrease in spiral angle.

It may be noted that this fibre, unlike cotton, contains about 15% lignin, which, as mentioned before, acts as a cementing material. Though lignin is absent in some strong fibres like ramie and cotton, it adds to the strength of the fibres if and where it is present. It is well known that lignin is removed on mercerisation and this is evident from our X-ray photographs (figures 5, 6, 7, 8) where we find the gradual fading, though not complete disappearance, of the two reflections which are ascribed to lignin. As lignin is supposed to be the cementing force, removal of it will naturally result into the loss of strength of the fibre.

That the crystallites are better oriented as a result of mercerisation is evident from the gradual decrease in the length of the (002) arcs, as can be seen in our mercerised patterns It is expected that this preferred orientation, i.e. steeper spirals would increase the strength of the fibre. But our findings show that the strength decreases. It seems that the increase in strength due to better orientation as a result of mercerisation has been outweighed by the loss of strength due to the removal of lignin in course of mercerisation. It, therefore, explains the seemingly contradictory findings as to why in spite of gradual decrease in intrinsic strength, the length of the arcs becomes gradually shorter.

It is also well known that the length of the micelle is one of the most important factors which go to build up the strength of fibres. Our investigation on the

determination of the length and width of the micelle (Laue, 1926) in raw and mercerised fibres is in progress. These results will be published shortly.

It may be pointed out that the single fibres which are used by local weavers here are coarse and this is rather a handicap to them so far as the use of this fibre in finer garments is concerned. Our optical study has revealed that these socalled single fibres get splitup into a number of finer fibrils under the action of very dilute acids. Investigation in this line is also in progress. In conclusion, it may be mentioned that if this particular type of leaf fibre, which is so abundant in Burma and also in India, along with some other members of the 'leaf fibre' group could be studied with the intensity and care it deserves, it is expected that there will be some more welcome additions in the family of ideal textile fibres.

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